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HYDROGEN EMBRITTLEMENT OF METAL FASTENERS DUE TO PHOSPHORIC ACID CONTAINMENT SYSTEM (PACS) EXPOSURE VOLUME 5 - DELIVERY ORDER 4, TASK 2



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The objective of this study was to determine if metal fasteners are susceptible to hydrogen embrittlement due to exposure to a phosphoric acid containment system (PACS) surface preparation. A PACS is used to anodize aluminum surfaces in preparation for adhesive bonding. Due to the containment system provided by PACS, it has the ability to be used in aircraft repair applications. Concerns have been raised as to whether the PACS surface preparation may be used in an area that contains metal fasteners.

The objective was accomplished by exposing mechanically loaded fasteners of various metal materials to PACS. Following the PACS exposure, the fasteners were rinsed and reloaded for 96 hours. Failure of the fasteners during either the PACS exposure or the sustained loading deemed the fastener embrittled.

#### 15. SUBJECT TERMS

Hydrogen Embrittlement, Metal Fasteners, Fastener Failures, PACS Surface Preparation, Fastener Test Methods

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#### **PREFACE**

This report covers the efforts performed by the University of Dayton Research Institute (UDRI), Dayton, OH 45469-0130, during the period from September 13, 1996 to September 30, 1997. The work was conducted under Air Force Contract F33615-95-D-5616, Delivery Order 0004 under the direction of Mr. James Folck (WL/MLSE) of Wright-Patterson Air Force Base, Ohio. Mr. Bill Schweinberg and Mr. Steve Adams of Warner-Robins Air Logistics Center (WR-ALC/TIEDD) were the Air Force Project Engineers.

The work described herein was conducted at UDRI in the Plastics, Adhesive, and Composite Group. The program was managed by Mr. D.R. Askins. Mr. W.B. Pinnell was the Principal Investigator. Additional contributions were made by Messrs. P. Muth, J. Stalter, and M. Pierson.

This report was submitted for publication in December 1997. The contractor's report number is UDR-TR-97-142.

### 1. OBJECTIVE

The objective of this program was to determine if metal fasteners are susceptible to hydrogen embrittlement due to exposure to a phosphoric acid containment system (PACS) surface preparation. This objective was to be accomplished by exposing fasteners of different metal materials to a PACS exposure while under a mechanical load. The exposed fasteners would then be rinsed and reloaded under a sustained load for 96 hours. If the fastener were to fail at any time during either the PACS exposure or the sustained loading, the fastener would be deemed embrittled.

#### 2. BACKGROUND

Concerns have been raised regarding the effects of acid prebond surface preparations on metal fasteners used on aircraft. The particular surface preparation process of concern is the PACS, which produces a phosphoric acid anodized surface. The advantage of the PACS is that the area which is anodized is controlled by a containment system. Therefore, PACS can be used on aircraft in areas where the anodization area cannot be controlled.

The effect of the phosphoric acid on any metal fasteners located in the anodized area is unknown. Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) contains hydrogen which may be absorbed into the metal, which in turn, may cause the metal fastener to become embrittled. Inappropriately applied fasteners (overtorqued) and/or extreme PACS exposure conditions (higher concentration, longer exposure time, higher temperature) may increase the chance of the embrittlement of the fasteners. Testing for embrittlement of the fasteners due to PACS exposure was performed in these worst-case scenarios.

### 3. TEST PROGRAM

3.1 Fasteners - The fasteners included in this program were chosen by WR-ALC. Each was a Hi-Lok (Hi-Shear Product) fastener with a 0.1875-inch grip diameter and a 1.00-inch grip length, Figure 1. The fasteners differed according to material (Ti 6Al 4V, H-11 Tool Steel, Alloy Steel, 431 Stainless, A-286 Steel), plating (aluminum, cadmium, nickel-cadmium, passivated), lubrication (cetyl alcohol), and fastener type (protruding head tension, protruding head shear). A list of the fasteners and information concerning the fasteners is shown in Table 1. The type of plating and lubrication chosen for the fasteners in this program was determined by their availability. The mechanical property data and fastener specifics shown in Table 1 were obtained in the Hi-Shear fastener drawings as well as the fastener lot material specifications/ certifications. The material modulus values were obtained from various metal handbooks. Fifty of each fastener type was purchased. All 50 came from the same manufacturing lot for that particular fastener.

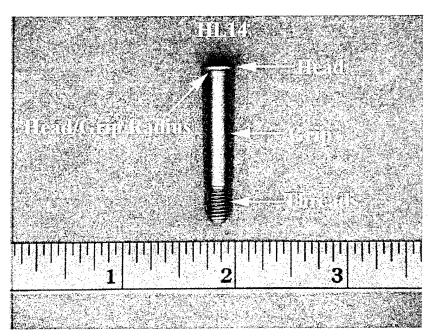


Figure 1. Overall Photo of Fastener HL14 (Shear Fastener). The tensile fasteners were similar except they contained a slightly larger head diameter than the shear fastener.

A certification sheet was available for each of the fasteners. This certification verified that each of the 50 fasteners met the design dimensions, strengths, and materials. The certification is based on a random number of fasteners tested from each lot. Since each and every fastener in a lot is not tested, an uncontrollable variable exists. This variable

Table 1. Fastener Information

				Minimum	Minimum	Minimum	
Fastener		Plating /	Head Style /	Strength <sup>1</sup>	Strength <sup>1</sup>	l ensile Load <sup>1</sup>	Material
Designation	Material	Lubrication	Application	(ksi)	(ksi)	(Ibs)	(Msi)
HL12VAZ-6-16		Aluminum/	Protruding /				
	Ti 6Al 4V	Cetyl Alcohol	Tension	160	95 <sup>2</sup>	3180	16.5
HL14-6-16	H-11 Tool	Ni-Cd/	Protruding /				
	Steel	Cetyl Alcohol	Shear	$260-280^3$	156	3700	29.0
HL20PB-6-16	Alloy Steel	Cadmium /	Protruding/				
		Cetyl Alcohol	Tension	160-180	954	3180	30.0
HL32PB-6-16	431	Cadmium /	Protruding /				
	Stainless	Cetyl Alcohol	Tension	Not	$125^{6}$ .	4350	29.0
	Steel			Available <sup>5</sup>			
HL40-6-16	A-286 Steel	Passivated /	Protruding/				
		Cetyl Alcohol	Shear	1607	95	2000	29.1

<sup>1</sup> Taken from fastener drawings and certifications

<sup>2</sup> Shear strength of an HL10 protruding shear fastener (same material as HL12)

<sup>3</sup> Tensile strength of an HL16 protruding tensile fastener (same material as HL14)

<sup>4</sup> Shear strength of an HL18 protruding shear fastener (same material as HL20)

<sup>5</sup> Tensile strength was not available for the HL32 protruding tensile fastener <sup>6</sup> Shear strength of an HI 30 protruding shear fastener (same material of HI 32)

<sup>6</sup> Shear strength of an HL30 protruding shear fastener (same material as HL32)

<sup>7</sup> Tensile strength of an HL48 protruding tensile fastener (same material as HL40)

was considered to be insignificant. Therefore, all fasteners in a manufacturing lot were considered to meet the design dimensions, strengths, and materials.

The HL14 fastener tested in this program was a substitute for HL16, which was originally chosen by WR-ALC. The substitute was necessary due to the unavailability of the HL16 fastener. The HL14 (shear type fastener) was chosen since it was the same material (H-11 Tool Steel) as the HL16 (tensile type fastener). Since the design of the shear fastener is slightly different (smaller head diameter) than the tensile fastener, the minimal tensile load given in the specification for HL14 was lower than the minimum load for the tensile fastener (HL16).

Two additional fasteners (HL102 and HL158) originally chosen by WR-ALC for testing were unavailable. Various fastener distributors indicated that these fasteners were no longer produced. As a result, WR-ALC decided to eliminate these two fasteners from the test program.

The fasteners were tested in two different conditions: (1) as-received (with plating and lubrication) and (2) stripped (no plating or lubrication). The plating was mechanically stripped from the fasteners with a steel wire wheel. The stripped condition simulated a fastener in the field that had some (or all) of its plating removed during service. Optical examinations of the fastener following the stripping indicated that all of the plating had been removed and no significant damage was present on the fastener. Detailed analysis of the stripped fasteners was not performed to validate the complete removal of the plating/lubrication or that no damage was inflicted. Therefore, the condition of each stripped fastener was not completely controlled.

The as-received fasteners were tested both with and without a PACS exposure. The as-received fasteners without a PACS exposure were tested to assure that they were not already embrittled due to poor manufacturing and/or sitting in storage. The stripped fasteners were only tested after a PACS exposure. Four of each type of fastener were tested in the three conditions described above. Failure of the fasteners in either the PACS exposure or the sustained loading deemed the fastener material type to be embrittled due to the PACS exposure.

**3.2** <u>Preloading of Fasteners Prior to PACS Exposure</u> – Prior to exposure to the PACS environment, each of the fasteners was preloaded (85 percent of the minimum tensile load of the fastener) in a manner similar to that described in the Elongation Method of MIL-

STD-1312-5A (Fastener Test Methods, Method 5 Stress Durability). Four fasteners of the same material were PACS exposed at the same time. Prior to the exposure, each of the four fasteners was elongated using a steel barrel fixture as shown in Figure 2. The loading and elongation measurements of the fasteners were performed at ambient conditions. The fastener was placed through the barrel and attached with a stainless steel nut. The length of the unloaded fastener was then measured with a flat end micrometer to 0.0001 inch. The nut was then tightened until the fastener reached a desired elongation. The desired elongation was determined by taking into account the minimum tensile load, the fastener material's modulus, the grip length, shank area, length of threads located between the top of the nut and the bottom of the grip, and the minimum thread area. These parameters are shown in Table 2 for each of the five fastener types. Table 2 also shows the equation (taken from MIL-STD-1312-5A) used to compute the desired elongation. Table 3 shows the actual fastener elongations measured during loading, the corresponding tensile loads, and percentages of the minimum tensile load.

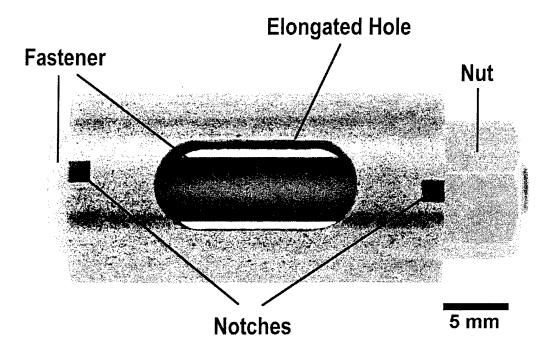


Figure 2. Photograph of the Fastener/Barrel Fixture. The stainless steel nut is tightened until the desired elongation of the fastener is achieved. The barrel contains elongated holes and notches to allow the acid to contact the fastener's grip, grip/head radius, and two threads below the grip.

Table 2. Calculated Elongation of Fasteners in Steel Barrels

			95% of				Loaded	i.	
		Minimum	Minimum	Material	Grip	Grip	Thread	Minimum	Desired
		Tensile	Tensile	Modulus	Length	Area	Length <sup>1</sup>	Thread	Elong. <sup>2</sup>
Fastener	Material	Load (lb)	Load (lb)	(Msi)	(in)	$(in^2)$	(in)	Area (in²)	(in)
HL12	Ti 6Al 4V	3,180	2,703	16.5		0.0275	0.07	0.0198	0.0065
HL14	H-11 Tool	3,700	3,145	29.0	Т	0.0275	0.07	0.0198	0.0043
HL20	Alloy	3,180	2,703	30.0	: -	0.0275	0.07	0.0198	0.0036
HL32	431	4,350	3,698	29.0	T	0.0275	0.07	0.0198	0.0051
HL40	A-286	2,000	1,700	29.1	1	0.0275	. 0.07	0.0198	0.0023

Thread length between end of grip and top of nut = length of barrel – grip length of fastener

Where: Length of barrel = 1.07 inch

Grip length of fastener = 1.00 inch

Where: Length of barrel = 1.07 inch
Grip length of fastener = 1.00 inch

Desired elongation = (85 percent load / modulus) x [(grip length / grip area) + (loaded thread length / minimum thread area)]

Table 3. Measured Fastener Elongation and Load in Steel Barrels

Fastener	Calculated Desired Elongation (in)	Actual Measured Elongation (in)	85% of Minimum Load (lb)	Calculated Actual Load <sup>1</sup> (lb)	Actual % of Minimum Load (%)
HL12	0.0065	0.0064	2,703	2,647	83.2
HL14	0.0043	0.0042	3,145	3,053	82.5
HL20	0.0036	0.0035	2,703	2,632	82.7
HL32	0.0051	0.0050	3,698	3,634	83.5
HL40	0.0023	0.0023	1,700	1,677	83.9

Calculated Actual Load = (modulus x measured elongation) / [(grip length / grip area) + (loaded thread length / thread area)]

The measurement of the fastener elongation was controlled in that it was performed with a calibrated micrometer. There were, however, several aspects of the elongation measurement process that could not be perfectly controlled. These included a slight difficulty in the actual length measurement and the slight variation ( $\pm$  3 °F) in day-to-day ambient lab temperature. The measurement difficulty derived from the inherent danger of the fasteners failing during the measurements and the small surface area at one end of the fastener in which one end of the micrometer was placed to measure the fastener's length.

The steel barrel shown in Figure 2 was machined from 4340 steel and hardened to 39-43 Rockwell C. A 0.25-inch-diameter hole was machined through the length of the barrel. This hole was large enough to accept the 0.1875-inch-diameter fastener. The diameter of the fastener head (0.357 - 0.377 inch tension fastener; 0.295 - 0.315 inch shear fastener) was large enough that the head was sufficiently supported by the barrel around the 0.25-inch-diameter hole. The hole diameter in the barrel was large enough such that the head/grip radius did not come in contact with the steel barrel. The barrel contained two elongated holes (one on each side) and two notches at each end. The elongated holes and notches provided a path for the acid to thoroughly wet the fastener at the grip, at the head/grip radius, and at the first two threads below the grip of the fastener. The length of the barrel was 1.07 inches. This length was used such that the 1.00-inch grip length plus two threads of the fastener would be under load and exposed to the PACS.

**3.3** PACS Exposure – The PACS exposure of the fasteners was performed while the fasteners were under load in the steel barrel fixture. An ATACS Model 0810 Phosphoric-

Acid Anodizer Containment System was used to conduct the PACS exposures. Various parameters associated with a normal PACS exposure were altered such that a worst-case scenario exposure was performed. The altered parameters included the temperature at which the exposure occurred (100 °F, as opposed to room temperature), the concentration of the phosphoric acid (15 percent as opposed to 12 percent), and the exposure time (1 hour as opposed to 25 minutes).

The test setup of the exposure is shown in Figure 3. The loaded fasteners were placed into an aluminum tray such that the barrels made contact with the aluminum. A nonconductive, nonwoven cloth material was placed on top of and between each barrel. A stainless steel screen was then placed on top of the nonwoven cloth. One additional layer of the nonwoven cloth was then placed on top of the steel screen. The anode connector from the PACS unit was attached to the aluminum tray while the cathode was attached to the stainless steel wire. A tube for the inflow of acid/water was located at one end of the tray, while a tube for the outflow of acid/water was located at the other end. A sealant material was applied around the top of the tray and around the acid/water inputs and outputs to seal the vacuum bag that was placed on top of the last nonwoven cloth layer. This aluminum tray was then placed inside an air circulating oven set at 100 °F.

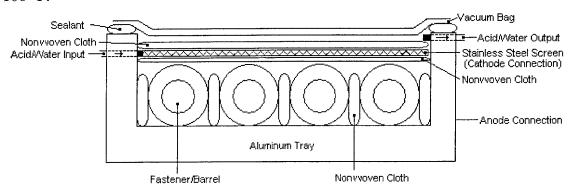


Figure 3. Schematic Drawing of the PACS Exposure Setup. The entire setup is placed inside an oven set at 100 °F. Drawing is not to scale.

The PACS unit was run at 10 Amps. The voltage was adjusted between 2.5 V and 6 V in order to maintain the 10 Amps. The acid/water flow rate was 80 cc/min. The 15 percent by weight phosphoric acid concentration was achieved by mixing 10 parts by volume 85 percent phosphoric acid with 90 parts by volume water. This ratio was extrapolated from the ratio given in ASTM D3933 for a 10 percent concentration (Preparation of Aluminum Surfaces for Structural Adhesives Bonding; Phosphoric Acid Anodizing). The

PACS runs consisted of a 1-hour flow of acid followed by a 10-20 minute flow of water. The water was used as a rinsing of the acid from the fasteners. Upon completion of the PACS exposure, the fasteners were unloaded from the barrels by removing the stainless steel nut. The fasteners were then towel-dried and tested under sustained loading (as described below).

The voltage and amperage were both controlled by the operator based on the indicator readings in the PACS unit. The time between unloading the fasteners from the barrels and application of the sustained loading was controlled to ensure that on every fastener, it was between 3-7 minutes. The 10-20 minute water rinse, while the fasteners were still loaded in the barrels, was conducted for each of the PACS exposures. However, no detailed examination was conducted to assure that all of the acid had been removed. Therefore, the amount of acid (if any) remaining on the fasteners was not controlled.

3.4 Sustained Loading - The sustained loading was performed in creep frames with specially designed fixtures to test the fasteners. A schematic drawing of the test setup is shown in Figure 4. Two fasteners were tested at one time in each creep frame. The two fasteners were placed in series such that the same load was applied to both. The steel fixtures (machined from tool steel) contained a side opening that the fastener and an insert could fit through. The insert (machined from tool steel) was placed inside the fixture. The insert contained a 0.2188-inch-diameter hole that the fastener would go through. The fastener was placed in the fixture and through the insert. The fastener was then threaded into a 0.75-inch-diameter steel all-thread which had been tapped to accept the 10-32 threads of the fastener, until only two threads of the fastener remained exposed. The series of all-threads, fixtures, and fasteners was connected to universal joints to aid in the alignment of the loading.

The creep frames were calibrated using a National Institute of Standards and Technology (NIST) calibrated load cell prior to any of the sustained loadings. Each frame contained a timer and a shutoff switch. The shutoff switch was used to turn off the timer in case of a fastener failure.

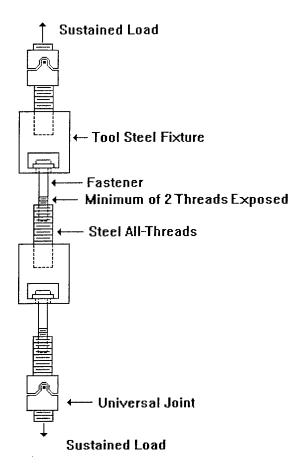


Figure 4. Schematic Drawing of the Sustained Loading Test Setup. The sustained load shown was applied by a creep frame. Drawing not to scale.

The sustained loading was performed on fasteners in three different conditions:

- (a) As-received with no exposure to PACS,
- (b) As-received with exposure to PACS, and
- (c) Stripped with PACS exposure.

The fasteners were loaded into the test fixtures as described above. All testing was performed at ambient conditions. The appropriate load level was then slowly applied to the fasteners. The loading was sustained until either the fastener failed or 96 hours elapsed, whichever came first.

The temperature and humidity of the room varied slightly ( $\pm$  3 °F,  $\pm$ 10 percent relative humidity) from day to day, thus these parameters were uncontrollable. These slight variations were deemed to be insignificant to the testing results.

### 4. FACTUAL DATA

Tables 4 through 8 summarize the results of the testing. Fasteners that failed during either the PACS exposure or the sustained loading were labeled as "Fail." Fasteners that did not fail during either the PACS exposure or the sustained loading were labeled as "Pass." Notes concerning the failures of the fasteners are also given in the tables.

Table 4. Testing Results of HL12

Fastener C	onditioning Prior to Sustain	ed Loading
As-Received (No PACS)	As-Received (PACS)	Stripped (PACS)
HL12-1Pass	HL12-9Pass	HL12-13Pass
HL12-2Pass	HL12-10Pass	HL12-14Pass
HL12-3Pass	HL12-11Pass	HL12-15Pass
HL12-4Pass	HL12-12Pass	HL12-16Pass

Table 5. Testing Results of HL14

Fastener C	Conditioning Prior to Sustain	ed Loading
As-Received (No PACS)	As-Received (PACS)	Stripped (PACS)
HL14-1Pass	HL14-5Fail <sup>1</sup>	HL14-9Fail <sup>3</sup>
HL14-2Pass	HL14-6Fail <sup>1</sup>	HL14-10Fail <sup>3</sup>
HL14-3Pass	HL14-7Fail <sup>1</sup>	HL14-11Fail <sup>3</sup>
HL14-4Pass	HL14-8Fail <sup>2</sup>	HL14-12Fail <sup>3</sup>

<sup>&</sup>lt;sup>1</sup> Failed at head during sustained loading. Failure occurred instantly upon load application. Contained no cracking in threads.

Failed in threads during PACS exposure. The length of time until failure was unable to be determined. Contained no cracking at the head.

<sup>&</sup>lt;sup>3</sup> Partial failure at head during PACS. The length of time until partial failure was unable to be determined. Complete failure at head occurred instantly upon application of the sustained load. Contained no cracking in the threads.

Table 6. Testing Results of HL20

Fastener Co	onditioning Prior to Sustain	ed Loading
As-Received (No PACS)	As-Received (PACS)	Stripped (PACS)
HL20-11Pass	HL20-16Pass	HL20-20Pass
HL20-12Pass	HL20-17Pass	HL20-21Pass
HL20-13Pass	HL20-18Pass	HL20-22Pass
HL20-14Pass	HL20-19Pass	HL20-23Pass

Table 7. Testing Results of HL32

Fastener Co	onditioning Prior to Sustain	ed Loading
As-Received (No PACS)	As-Received (PACS)	Stripped (PACS)
HL32-1Pass	HL32-5Fail <sup>1</sup>	HL32-9Fail <sup>2</sup>
HL32-2Pass	HL32-6Fail <sup>1</sup>	HL32-10Fail <sup>2</sup>
HL32-3Pass	HL32-7Fail <sup>1</sup>	HL32-11Fail <sup>1</sup>
HL32-4Pass	$HL32-8Fail^2$	HL32-12Fail <sup>2</sup>

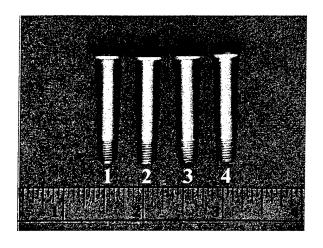
<sup>&</sup>lt;sup>1</sup> Failed at head during sustained loading. Failure occurred instantly upon sustained load application. Contained cracking in the threads.

Table 8. Testing Results of HL40

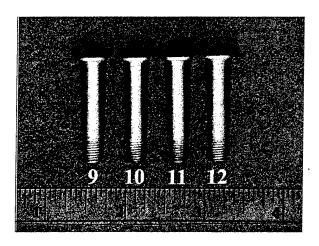
Fastener Conditioning Prior to Sustained Loading		
As-Received (No PACS)	As-Received (PACS)	Stripped (PACS)
HL40-1Pass	HL40-5Pass	HL40-9Pass
HL40-2Pass	HL40-6Pass	HL40-10Pass
HL40-3Pass	HL40-7Pass	HL40-11Pass
HL40-4Pass	HL40-8Pass	HL40-12Pass

**4.1** <u>As-Received No PACS Exposure</u> - All of the as-received fasteners that had not been exposed to PACS passed the sustained load test. Overall photos of these tested fasteners are shown in Figures 5 through 9. These test results showed that none of the fasteners were embrittled in the as-received condition. Thus, any failures occurring during the PACS exposure or sustained loading could be attributed to the exposures.

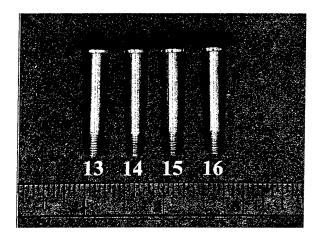
<sup>&</sup>lt;sup>2</sup> Failed in threads during sustained loading. Failure occurred instantly upon sustained load application. Contained cracking at the head.



HL12 As-Received No PACS Exposure



HL12 As-Received PACS Exposure



HL12 Stripped PACS Exposure

Figure 5. Overall Photos of the Tested HL12 Fasteners. No failures occurred at any of the test conditions.

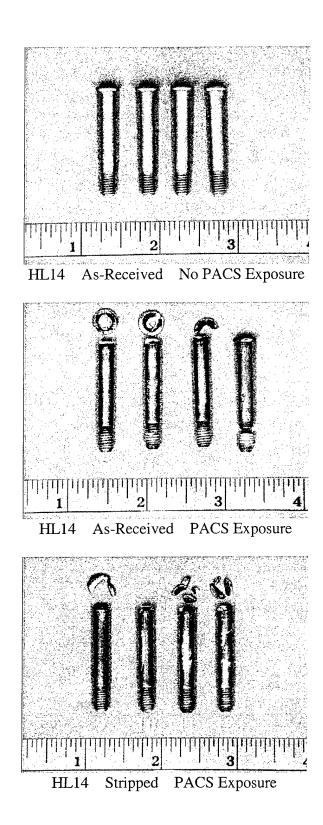
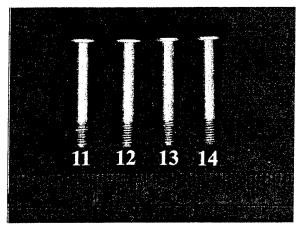
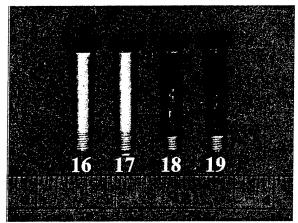


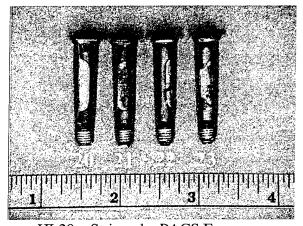
Figure 6. Overall Photos of the Tested HL14 Fasteners. All of the fasteners exposed to PACS failed at either the head (7 each) or in the threads (1 each).



HL20 As-Received No PACS Exposure

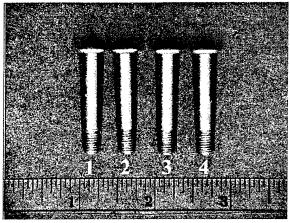


HL20 As-Received PACS Exposure

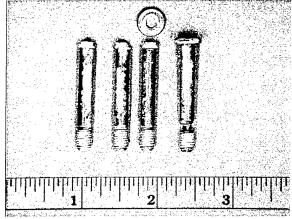


HL20 Stripped PACS Exposure

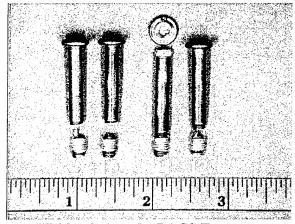
Figure 7. Overall Photos of the Tested HL20 Fasteners. No failure occurred at any of the test conditions.



HL32 As-Received No PACS Exposure

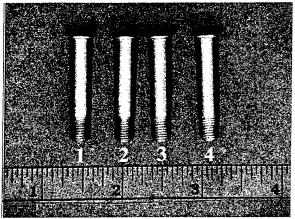


HL32 As-Received PACS Exposure

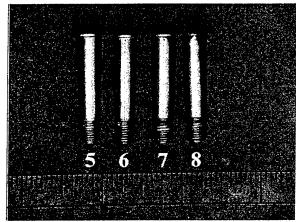


HL32 Stripped PACS Exposure

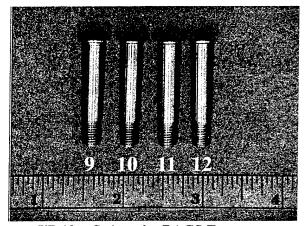
Figure 8. Overall Photos of the Tested HL32 Fasteners. All fasteners exposed to PACS failed at either the head (4 each) or within the threads (4 each).



HL40 As-Received No PACS Exposure



HL40 As-Received PACS Exposure



HL40 Stripped PACS Exposure

Figure 9. Overall Photos of the Tested HL40 Fasteners. No failures occurred in any of the test conditions.

4.2 <u>As-Received PACS Exposure</u> - Overall photos of all the as-received fasteners that had been exposed to PACS and tested under a sustained load are shown in Figures 5 through 9. Fasteners HL14 and HL32 both failed the testing either during the PACS exposure or sustained loading. The fasteners that failed in the sustained loading did so in the first few seconds after the load was applied. In these cases, one of the two fasteners in each creep frame failed first. The second fastener was then reloaded immediately in the same creep frame. Within the first few seconds of the reloading, the second fastener failed. The time it took for the fastener to fail in the PACS exposure was unable to be determined. Fasteners HL12, HL20, and HL40 did not fail during either the PACS exposure or the sustained loading.

All but one of the HL14 fasteners failed at the head during the sustained loading. Figure 10 shows an increased magnification photo of the fracture surface of the HL14-6 fastener that failed at the head. It was observed that much of the fracture surface was black in color. It is believed that this black area was caused by the phosphoric acid during the PACS exposure. Since the HL14 as-received with no PACS exposure fasteners did not fail and did not contain cracks either in the threads or around the head, it would appear that the cracks developed during the PACS exposure. One HL14 fastener failed within the threads during the PACS exposure. Much like the failure at the heads, the fracture surface was blackened due to the phosphoric acid.

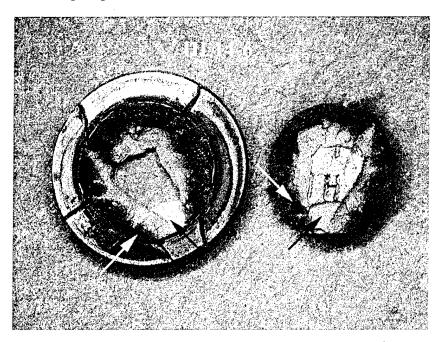


Figure 10. Increased Magnification of the Mating Fracture Surfaces of Fastener HL14-6's Head Failure. The white arrows identify a blackened fracture surface (believed to be caused by acid penetration into a crack). A clean metallic fracture surface (no acid penetration) is shown by the black arrows.

In the case of the HL32 fasteners, three failures occurred at the head and one in the threads. The fasteners that failed at the head, Figure 11, also displayed cracking in the threads, Figure 12. The reverse was also true (i.e., the fastener that failed in the threads also displayed cracking at the head), as shown in Figures 13 and 14. No blackened areas were noted on the fracture surfaces of the HL32 fasteners. Therefore, it was believed that no cracking existed prior to the sustained loading.

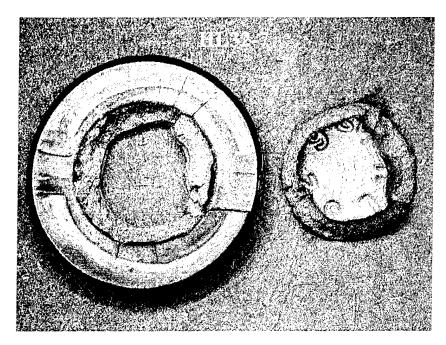


Figure 11. Increased Magnification of the Mating Fracture Surfaces of Fastener HL32-7's Head Failure.

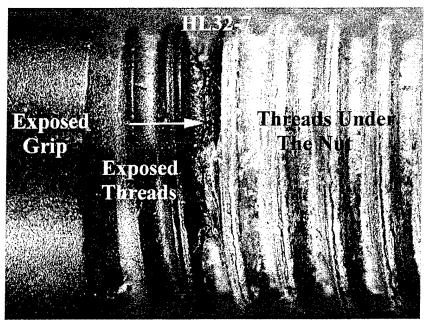


Figure 12. Increased Magnification of Cracking in the Threads of Fastener HL32-7. Arrow identifies the cracking located in the exposed/loaded threads.

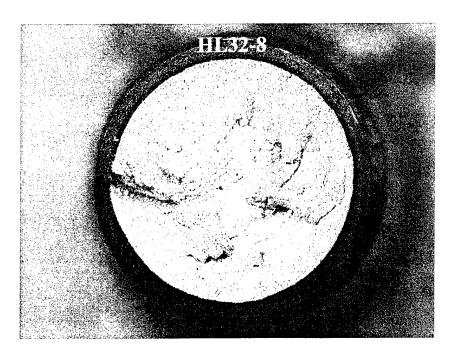


Figure 13. Increased Magnification of a Failure in the Threads of Fastener HL32-8.

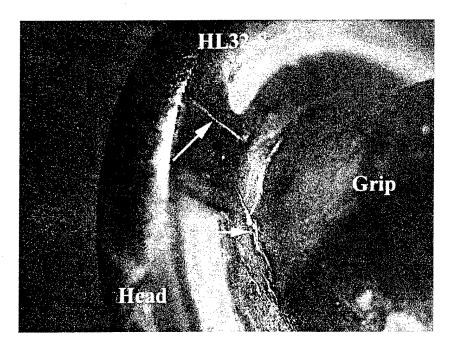


Figure 14. Increased Magnification of Cracking at the Head of Fastener HL32-8. The white arrows indicate cracking seen at the head and at the head/grip radius.

**4.3** Stripped PACS Exposure - Overall photos of all of the stripped fasteners that were exposed to PACS and tested under a sustained load are shown in Figures 5 through 9. Similar to the as-received (PACS exposure) fasteners, HL14 and HL32 failed within seconds of the application of the sustained load. Fasteners HL12, HL20, and HL40 did not fail during either the PACS exposure or the sustained loading.

The HL14 fasteners partially failed at the heads during the PACS exposure. The fasteners then completely failed at the head within the first few seconds of the sustained loading. These fasteners showed no signs of cracking in the threads. Once again, the fracture surfaces of the HL14 fasteners displayed areas of black discoloration (believed to be acid penetration during the PACS exposure).

The HL32 fasteners all failed within the first few seconds of sustained loading. Three of these fasteners failed within the threads while one failed at the head. Once again, the fasteners that failed at the head also exhibited cracking in the threads and vice versa.

#### 5. CONCLUSIONS

Based upon the results obtained in this test program, the following conclusions have been made:

- 1. The PACS exposure does appear to embrittle certain types of fastener materials including H-11 Tool Steel and 431 Stainless Steel. The fasteners fabricated from the higher strength materials, HL14 and HL32, were the ones that displayed a tendency to become embrittled. Literature shows that the degree of hydrogen embrittlement depends greatly on the strength level of the steel; resistance to hydrogen embrittlement decreases as the strength level is increased [1]. The results from this program are consistent with the behavior described in the literature.
- 2. The fastener with the highest strength (HL14) appeared to have initially failed during the PACS exposure. Thus, it appears as if this fastener/material is the most susceptible to embrittlement.
- 3. The stripping of the plating on the fasteners appeared to have little (if any) effect on the fastener's susceptibility to become embrittled. The same fasteners (HL14 and HL32) that failed in the stripped condition also failed in the as-received condition. Additionally, the same fasteners that were plated and had passed the exposure and sustained loadings also passed the same tests after they were stripped of their coating.

## 6. RECOMMENDATIONS

The results of this program showed that PACS does have the ability to embrittle at least some types of metal fasteners/materials. Therefore, PACS techniques should be investigated that would isolate the fasteners from the acid during the surface preparation.

## 7. REFERENCES

[1] Metals Handbook Desk Edition, American Society for Metals, 1985.